METHOD 7421

LEAD (ATOMIC ABSORPTION, FURNACE TECHNIQUE)

1.0 SCOPE AND APPLICATION

1.1 See Section 1.0 of Method 7000.

2.0 SUMMARY OF METHOD

2.1 See Section 2.0 of Method 7000.

3.0 INTERFERENCES

3.1 See Section 3.0 of Method 7000 if interferences are suspected.

3.2 Background correction is required.

3.3 If poor recoveries are obtained, a matrix modifier may be necessary. Add 10 uL of phosphoric acid (Paragraph 5.3) to 1 mL of prepared sample in the furnace sampler cup and mix well.

4.0 APPARATUS AND MATERIALS

4.1 For basic apparatus, see Section 4.0 of Method 7000.

4.2 Instrument parameters (general):

4.2.1 **Drying time and temp:** 30°sec at 125°C.
4.2.2 **Ashing time and temp:** 30°sec at 500°C.
4.2.3 **Atomizing time and temp:** 10 sec at 2700°C.
4.2.4 **Purge gas:** Argon.
4.2.5 **Wavelength:** 283.3 nm.
4.2.6 **Background correction:** Required.
4.2.7 Other operating parameters should be set as specified by the particular instrument manufacturer.

NOTE: The above concentration values and instrument conditions are for a Perkin-Elmer HGA-2100, based on the use of a 20-uL injection, continuous-flow purge gas, and nonpyrolytic graphite. Smaller sizes of furnace devices or those employing faster rates of atomization can be operated using lower atomization temperatures for shorter time periods than the above-recommended settings.

5.0 REAGENTS

5.1 See Section 5.0 of Method 7000.

5.2   Preparation of standards:

    5.2.1 **Stock solution:** Dissolve 1.599 g of lead nitrate, $Pb(NO_3)_2$ (analytical reagent grade), in Type II water, acidify with 10 mL redistilled HNO_3, and dilute to 1 liter with Type II water. Alternatively, procure a certified standard from a supplier and verify by comparison with a second standard.

    5.2.2 Prepare dilutions of the stock solution to be used as calibration standards at the time of analysis. The calibration standards should be prepared using the same type of acid and at the same concentrations as in the sample after processing (0.5% v/v HNO_3).

    5.3   Phosphoric acid: Reagent grade.

6.0   SAMPLE COLLECTION, PRESERVATION, AND HANDLING

    6.1   See Chapter Three, Section 3.1.3, Sample Handling and Preservation.

7.0   PROCEDURE

    7.1   Sample preparation: The procedures for preparation of the sample are given in Chapter Three, Section 3.2.

    7.2   See Method 7000, Paragraph 7.3, Furnace Procedure. The calculation is given in Method 7000, Paragraph 7.4.

8.0   QUALITY CONTROL

    8.1   See Section 8.0 of Method 7000.

9.0   METHOD PERFORMANCE

    9.1   Precision and accuracy data are available in Method 239.2 of Methods for Chemical Analysis of Water and Wastes.

    9.2   The performance characteristics for an aqueous sample free of interferences are:

    Optimum concentration range: 5-100 ug/L.
    Detection limit: 1 ug/L.

    9.3   The data shown in Table 1 were obtained from records of state and contractor laboratories. The data are intended to show the precision of the combined sample preparation and analysis method.

10.0 REFERENCES

1.   Lead by Flameless Atomic Absorption with Phosphate Matrix Modification, Atomic Spectroscopy, $\underline{1}$ (1980), no. 3, pp. 80-81.

2.   Gaskill, A., Compilation and Evaluation of RCRA Method Performance Data, Work Assignment No. 2, EPA Contract No. 68-01-7075, September 1986.

TABLE 1. METHOD PERFORMANCE DATA

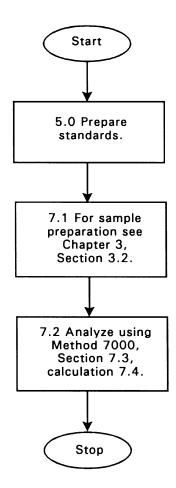| Sample Matrix	Preparation Method	Laboratory Replicates
Contaminated soil	3050	163, 120 mg/g
Paint primer	3050	0.55, 0.63 mg/g
Lagoon soil	3050	10.1, 10.0 ug/g
NBS SRM 1646 Estuarine sediment	3050	23.7 ug/g[a]
NBS SRM 1085 Wear metals in lubricating oil	3030	274, 298 ug/g[b]
Solvent extracted oily waste	3030	9, 18 ug/L

[a]Bias of -16% from expected.

[b]Bias of -10 and -2% from expected, respectively.

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                        (    Start    )
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                    ┌──────────────────────┐
                    │    5.0 Prepare        │
                    │    standards.         │
                    └──────────┬───────────┘
                               │
                               ▼
                    ┌──────────────────────┐
                    │   7.1 For sample      │
                    │  preparation see      │
                    │    Chapter 3,         │
                    │    Section 3.2.       │
                    └──────────┬───────────┘
                               │
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                    ┌──────────────────────┐
                    │  7.2 Analyze using    │
                    │   Method 7000,        │
                    │    Section 7.3,       │
                    │  calculation 7.4.     │
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                        (    Stop     )
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